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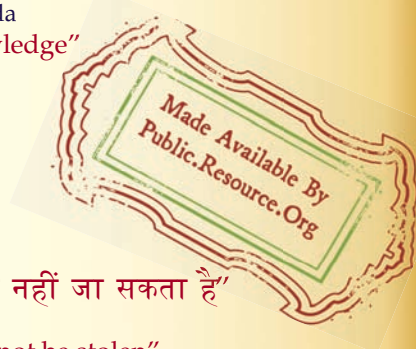
IS 13590 (1992): Acetoacet-2-anisidide [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”

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IS 13590 : 1992

(Reaffirmed 2002)

भारतीय मानक

ऐसीटोऐसीट-2-ऐनिसिडाइड — विशिष्ट

Indian Standard

ACETOACET-2-ANISIDIDE—SPECIFICATION

UDC 667.284.1

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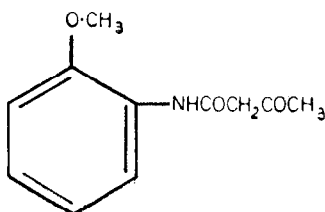
December 1992

Price Group 2

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Acetoacet-2-anisidide ($C_{11}H_{13}NO_3$) is an important dye intermediate used in the manufacture of yellow organic pigments. It has the following structural formula:



ACETOACET-2-ANISIDIDE
(Molecular Mass 207.2)
CAS Registry Number [92-15-9]

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ACETOACET-2-ANISIDIDE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for acetoacet-2-anisidide.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2552 : 1979	Steel drums (galvanized and ungalvanized) (<i>second revision</i>)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white crystalline powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1979) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Indicating the source of manufacture;
- c) Tare, net and gross mass; and
- d) Batch number.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING AND CRITERIA FOR CONFORMITY

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Each individual sample shall be tested for melting range and assay.

5.2.2 Test for description and the remaining characteristics, namely 2-anisidine content and matter insoluble in sodium hydroxide solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 For Individual Samples

The lot shall be declared as conforming to the requirement of melting range and assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 For Composite Samples

For declaring the conformity of the lot to the requirement of description and all other characteristics tested on the composite sample (*see* 5.2.2), the test results for each of the characteristics shall satisfy the relevant requirements given in 3.1 and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the method prescribed in IS 5299 : 1969 and Annex A as indicated in col 4 and 5 Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Acetoacet-2-anisidide
(*Clause 3.2*)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Cl No. in IS 5299 : 1969	Annex of This Standard
(1)	(2)	(3)	(4)	(5)
i)	Melting range, °C	83 to 85	8	—
ii)	Assay, percent by mass, <i>Min</i>	98	—	A-1
iii)	2-Anisidine content, percent by mass, <i>Max</i>	0.2	—	A-2
iv)	Matter insoluble in sodium hydroxide solution, percent by mas, <i>Max</i>	0.2	—	A-3

ANNEX A

(Table 1 and Clause 6.1)

METHODS OF TEST FOR ACETOACET-2-ANISIDIDE

A-1 ASSAY

A-1.0 Outline of the Method

The solution of the materials titrated against standard diazonium solution.

A-1.1 Reagents

A-1.1.1 Hydrochloric Acid — 30 percent (*m/v*).

A-1.1.2 Sodium Nitrite Solution — 10 percent (*m/v*).

A-1.1.3 4-Nitroaniline — 99 percent, pure.

A-1.1.4 Sodium Hydroxide Solution — 40 percent (*m/v*).

A-1.1.5 Dilute Acetic Acid — 25 percent (*m/v*).

A-1.1.6 Sodium Acetate Solution — 20 percent (*m/v*).

A-1.1.7 H-Acid Indicator Solution — Dissolve 0.5 g of H-acid in 100 ml of water containing 1 g of soda ash.

A-1.1.8 Acetoacet-2-Anisidide — Reference sample of known purity.

A-1.1.9 Tetrazodianisidine Solution

Weigh accurately about 2 g of dianisidine base in a beaker and dissolve in 7 ml of hydrochloric acid (heat, if necessary, up to 50°C). Cool with ice at about 0°C and add immediately 12 ml of sodium nitrite solution and make up the volume to 100 ml with ice cold water, shake mildly. Test for excess nitrite with starch-iodide test paper. Store this solution in an amber coloured bottle in a cool place.

A-1.2 Procedure

A-1.2.1 Preparation of Diazonium Solution of 4-Nitroaniline

Weigh accurately 2 g of 4-nitroaniline in a 250-ml glass beaker containing 5 ml of hydrochloric acid and 100 ml of water, heat up to 60°C to dissolve completely. Cool externally to 0 to 50°C with ice. Then add 10 ml of sodium nitrite solution with stirring. Test for excess of sodium nitrite by spotting on starch-iodide test paper. Allow to stand for 30 minutes and then repeat the above test. Filter to remove insoluble matter and then make up the filtrate to 200 ml in a volumetric flask. Store the solution in an ice bath in the dark.

A-1.2.2 Standardization of Diazonium Solution

Weigh accurately 0.751 g of acetoacet-2-anisidide of known purity and dissolve with 1 ml of sodium hydroxide solution in 25 ml water. Dilute the solution to about 300 ml with water. Add dropwise dilute acetic acid while stirring till pH is 7, which may be judged by using pH paper. Add to the suspension 100 ml of sodium acetate solution.

Add the diazonium solution while stirring mechanically from a burette equipped with jacket through which water is circulating at about 10°C. The burette should be amber glass to minimize any decomposition of the diazonium solution by light.

A-1.2.3 Titrate as rapidly as spot test permits. Test for excess acetoacet-2-anisidide by placing a few drops of titration mixture on a filter paper. Place a few drops of tetrazodianisidine solution about one centimetre away from the edge of the liquid mark. Brown colour develops where the two liquid portions meet on the filter paper, if excess of acetoacet-2-anisidide is present. Similarly, try with H-acid indicator to test the excess diazonium solution. If a pink colour develops at the junction, excess of diazonium solution is indicated. Initially, add diazonium solution in 1 to 2 ml portions testing titration mixture after each addition for excess of acetoacet-2-anisidide and diazonium salt. Add diazonium standard solution in portion of 0.2 ml as the end point approaches. The end point is reached when no reddish brown colour is given with tetrazodianisidine solution and no pink colour or very faint pink colour is given with H-acid indicator. Note the volume of diazonium solution required for titration.

A-1.2.4 Repeat the titration as prescribed in A-1.2.3 using the test sample of acetoacet-2-anisidide.

A-1.3 Calculation

$$\text{Normality of diazonium solution, } N = \frac{P \times 0.751}{V \times 20.72}$$

where

P = purity (percent by mass) of the known sample; and

V = volume, in ml, of diazonium solution required for the known sample.

$$\text{A-1.3.2 Assay, percent by mass} = \frac{V_1 \times N \times 20.72}{0.751}$$

where

V_1 = volume, in ml, of diazonium solution required for the test sample; and

N = normality of diazonium solution.

A-2 DETERMINATION OF 2-ANISIDINE

A-2.0 Outline of the Method — 2-Anisidine is determined by thin layer chromatography.

A-2.1 Apparatus

A-2.1.1 Thin Layer Chromatographic Chamber — 25 cm × 25 cm.

A-2.1.2 Micropipette — 10 microlitre capacity.

A-2.1.3 Thin Layer Chromatography Plates — size 20 cm × 20 cm.

A-2.2 Reagents

A-2.2.1 2-Anisidine

Pure (reference sample). Prepare 0.001 percent, 0.002 percent, 0.004 percent solution in ethanol.

A-2.2.2 Eluent — chloroform.

A-2.2.3 Nitrous Fumes Chamber

A glass chamber of size 25 cm × 25 cm with a beaker of a capacity 100 ml containing approximately 50 ml of concentrated HCl (30 percent, *m/v*). The nitrous fumes are generated by adding a little solid sodium nitrite every time before inserting the plate.

A-2.2.4 Spray Reagent

0.5 percent solution of American Base (N-1-Naphthyl-ethylene diamine dihydrochloride) in methanol (*m/v*).

A-2.3 Procedure

Weigh accurately about 2 g of the sample (100 percent basis) and dissolve in 100 ml methanol. Take a plate coated with silica gel G and spot 10 microlitres of each of the test solution and reference solution along the start line at a distance of 3 cm from one another. Saturate the chromatographic chamber with the eluent. Insert the plate in the chamber and allow to run for about 25-30 minutes during which time the solvent front travels approximately 10 cm past the start line. Remove the plate from the chamber. After drying put the plate in the nitrous fumes chamber for about 30 seconds, remove the excess nitrous fumes by blowing air on it. Now spray the plate with American Base when the spots (red violet) characteristics of 2-anisidine become visible at R_f value of 0.35.

A-2.4 Reporting

Estimate the quantities of the impurity by comparing the colour intensity of the spot of the material under test with those of the solutions of the reference sample.

A-3 TEST FOR SOLUBILITY IN SODIUM HYDROXIDE SOLUTION

A-3.1 Reagent

A-3.1.1 Sodium Hydroxide Solution — 1 N.

A-3.2 Procedure

Weigh 1 g of the material into a test tube. Add exactly 10 ml of sodium hydroxide solution to it and stir with a glass rod. Dissolve the material completely at room temperature. Filter through a sintered crucible of porosity G 4, wash thoroughly, dry, weigh and calculate the percentage matter insoluble as given in 10.2 of IS 5299 : 1969.

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Doc : No. PCD 11 (912)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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